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Nanotechnologies — Sample preparation for the characterization of metal and metal-oxide nano-objects in water samples

Nanotechnologies — Préparation des échantillons pour la caractérisation de nano-objets métalliques et d'oxydes métalliques dans les échantillons d'eau

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Foreword

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This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

With the increasing use of manufactured nano-objects in commercial products and applications, such as consumer and healthcare products, solar panels, batteries, surface coatings, and water treatment, it is likely that these nano-objects will eventually be released to the environment, especially in aquatic environments. There are, however, limited technical data available on the occurrence/transport/fate of manufactured nano-objects after they are released to the aquatic environment. Together with the current global shortage of water supply and an increasing demand for water recycling, concerns for the potential health impacts of manufactured nano-objects in water will increase.

Related to nano-objects in aqueous matrices, knowledge of environmental parameters like natural organic matter content, pH, ionic strength (IS) etc., is important since these may influence particle size, fate, stability and chemical composition. An aqueous sample can be a complex mixture of particles of different nature, size, reactivity, composition, agglomeration state and shape. Hence the initial preparation of the samples, such as pre-treatment and size fractionation, are critical steps for any subsequent analysis of the nano-objects. A consolidated table listing common fractionation techniques is given by Simonet, et al.[1] and Hassellov, et al.[2].

Although several methods for the detection and characterization of manufactured nano-objects in aqueous matrices are described in ISO/TR 18196:2016, the methods are at various stages of development into technical specifications or standards. Most importantly, there is no accepted standard as yet on pre-analysis treatment (i.e. collection, storage and size fractionation) of manufactured nano-objects in water. This document can contribute to the development of a future international standard for the analysis and characterization of metal and metal-oxide nanoparticles in aqueous matrices. This will allow interlaboratory comparison of results and contribute to future studies of commercial products containing manufactured nano-objects, thus, finally, support the growth of nanotechnology related industries.

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Nanotechnologies — Sample preparation for the characterization of metal and metal-oxide nano-objects in water samples

1 Scope

This document provides an overview of approaches of sample preparation (i.e. pre-treatment and size-fractionation) for analytical measurements applied to surface and drinking water, potentially containing relevant amounts and types of metal and metal oxide nano-objects, including collection from source and storage of samples, pre-concentration of analytes, and their fractionation.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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ISO and IEC maintain terminological databases for use in standardization at the following addresses:

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— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

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3.1
manufactured nanomaterial
nanomaterial intentionally produced to have selected properties or composition

[SOURCE: ISO/TS 80004-1:2015, 2.9]

3.2
measurand
quantity intended to be measured

[SOURCE: ISO/IEC Guide 99:2007, 2.3, modified — The notes to entry have been deleted.]

3.3
nano-object
discrete piece of material with one, two or three external dimensions in the nanoscale

Note 1 to entry: The second and third external dimensions are orthogonal to the first dimension and to each other.

[SOURCE: ISO/TS 80004-2: 2015, 2.2]

3.4
nanostructured material
material having internal nanostructure or surface nanostructure

Note 1 to entry: If external dimensions are in the nanoscale, the term *nano-object* (3.2) is recommended.

[SOURCE: ISO/TS 80004-1:2015, 2.7, modified — Note 1 to entry has been replaced.]

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3.5
surface water
water which flows over, or rests on, the surface of a land mass

[SOURCE: ISO 13164-1:2013, 3.1.20]

3.6
drinking water
water either in its original state or after treatment, intended for drinking, cooking, food preparation, or other domestic purposes, regardless of its origin

Note 1 to entry: Also known as potable water.

[SOURCE: ISO 5667-5:2006, 2.2, modified]

4 Symbols and abbreviated terms

HDPE	high density polyethylene
FFF	field-flow fractionation
Flow FFF	flow field-flow fractionation
UF	ultrafiltration
SEC	size exclusion chromatography
GPC	gel permeation chromatography
SDS	sodium dodecyl sulfate
DLS	dynamic light scattering
UV-vis	ultraviolet–visible spectroscopy
RC	regenerated cellulose
PES	polyethersulfone

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5 Types of metal and metal oxide-based manufactured nano-objects

Based on the relevance of existing commercial products[1)], the following metal and metal oxide-based nano-objects were considered in the development of this document:

— Titanium dioxide (TiO_2): One of the most commonly used nanomaterial ingredients in many common consumer products (e.g. sunscreens, antifungal paints) and medical products;

— Zinc oxide (ZnO): Also widely used in sunscreens and medical products;

— Silver (Ag): Commonly used as an antibacterial agent in consumer textiles and medical products;

— Gold (Au): Widely used in biomedical applications, including bioassays, drug delivery and hyperthermal therapy.

6 Types of water matrices of interest

Two types of water matrices were considered in this document, surface water and drinking water. Surface water is the primary water source receiving waste streams, and thus it is prone to receive

1) Industry-wide applications: http://www.understandingnano.com/nanoparticles.html.

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manufactured nano-objects used in various commerical products. Drinking water in urban cities is produced by water treatment plants to remove all particulates which can include nano-objects. However, Kaegi and coworkers have found trace quantities of nano-objects in water samples after treatment[13]. Thus, with increasing use of manufactered nano-objects in commercial products, it is likely that monitoring of nano-objects in drinking water will become important in the future.

7 Sample collection and storage

7.1 General

To minimize changes in the state of water samples from the point of sample collection to analysis, precaution should be taken during collection, transport and storage. Currently there is no standard in collection, transport and storage of surface water samples containing nano-objects, but the following ISO publications on water quality – Sampling, should be used as a starting point for the development of nano-object specific procedures:

— ISO 5667-1 on guidance on the design of sampling programmes and sampling techniques;

— ISO 5667-3 on preservation and handling of water samples as well as the use of sample containers;

— ISO 5667-4 on guidance on sampling from lakes;

— ISO 5667-5 on guidance on sampling of drinking water from treatment works and piped distribution systems;

— ISO 5667-6 on guidance on sampling of rivers and streams.

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Since nano-objects in surface water may undergo continuous physicochemical changes, it is useful to carry out the analysis of the samples with minimal delay.

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7.2 Containers for sample collection and storage

Besides taking guidance from ISO 5667-3, common types of containers used in collection and storage of freshwater samples for nano-object analysis include high density polyethylene (HDPE)[8][9][10] and borosilicate glass[11]. The containers, especially HDPE, are typically pre-washed with dilute acid before use. For physicochemical analysis of nano-objects, the collected samples are typically stored at ambient or sub-ambient temperatures but without freezing. Very often, water samples require stabilization and addition of anti-microbial agents to allow storage.

8 Sample pre-treatment

8.1 Introduction

Raw water samples collected from an industrial or environmental source often require pre-treatment to (i) remove large particulates and/or (ii) concentrate the nano-objects of interest. Together with dilution techniques (not discussed here), these are the main pre-treatment methods before the sample can be used for further size fractionation and/or analysis.

WARNING — Since nano-objects can adsorb onto large particulates, the following pre-treatment methods may potentially remove nano-objects to some extent.

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